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Original Article

EXPERIMENTAL INVESTIGATION AND ANALYSIS OF MECHANICAL PROPERTIES OF POLYVINYL ESTER/GLASS FIBER COMPOSITE WITH ALUMINA (AL_2O_3), MOLYBDENUM DISULFIDE (MOS_2) AND TITANIUM OXIDE (TIO_2) FILLERS

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ABSTRACT

In the present research work, the experimental analysis was conducted on Polyvinyl ester/Glass Fiber composite with Alumina (Al_2O_3), Molybdenum disulfide (MoS_2) and Titanium oxide (TiO_2) fillers. Polymer composites offer numerous advantages for the high strength to weight ratio due to low density, high modulus, and better stiffness. Low strength and ductility of Polyesters, vinyl-esters, and epoxy cannot be applied for technical applications, hence reinforced by fiber and fillers to form composite matrices. The increase in % of filler has enhanced the ultimate strength monotonously except for 12.5% of Tio_2 filler in the polyvinyl ester/GF composites. The addition of Al_2O_3 filler has better Young's modulus compared to other fillers and maximum Young's modulus was observed for 12.5% Al_2O_3 and there is a drastic increase in Young's modulus to almost 40% compared with 7.5% Al_2O_3 polyvinyl ester composite. Flexural strength of particulate filled polyvinyl ester composite composites improved due to strong interfacial adhesion and the surface roughness act as mechanical interlocking. It is also evident that the addition of MoS_2 filler is more beneficial than that of Al_2O_3 filler in improving the flexural strength and modulus of polyvinyl ester/GF composite. The SEM study was conducted to infer the results obtained for the tensile and flexural tests conducted for the polyvinyl ester/GF composite.

KEYWORDS: Polyvinylester Polymer, Glass Fibre with Filler Composites, Al2O3 Filler, MoS2 Filler & TiO2 Filler

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INTRODUCTION

Polymer composites offer numerous advantages for the high strength to weight ratio due to low density, high modulus and better stiffness [1, 2]. Epoxy and polyesters are the popularly used thermosetting resins which are widely used in bulk applications for reinforced composites [3, 4]. They are generally formed by a condensation reaction between a glycol and an unsaturated dibasic acid. Low strength and ductility of Polyesters, vinyl-esters, and epoxy cannot be applied for technical applications, hence reinforced by fiber and fillers to form composite matrices [5]. If chemical resistance is the most important point of view vinyl esters are the prime choice [6]. The glass fibers, especially E and S type, as reinforcing materials used in fibre reinforced thermoset composites due to their length to stiffness and are economical [7, 8]. Vinyl Ester (VE) resins are produced from epoxy resins and unsaturated monocarboxylic acids. Their low room temperature viscosity coupled with rapid curing and relatively

low cost are promoting factors for molding processes [9, 10]. The glass fibers, especially E and S type, as reinforcing materials are the most widely used in fiber reinforced thermoset composites as they have enhanced mechanical and economical properties [11, 12]. In addition, vinyl ester resins possess high chemical and solvent resistance.

Madhusudhan T, et al., [13] have developed and subjected the composites to Mechanical Characterization of Jute and Rubber Particles Reinforced Epoxy Polymer Composites. In this tensile and flexural strength enhances proportionately with the fiber composition and are less influenced by rubber particulate as compared to fibers. The combination of these materials in composites can be used as an alternative in any synthetic fiber filled polymer composites. Madhusudhan T, et al [14] investigation on the Wear Resistance of Silica carbide particulate filler hybrid Composites reveals that the polymer composites with 10% sic show least wear loss. Madhusudhan T, et al [14] made Investigation on Wear Behavior of Sic particulate filler Hybrid Composites by Taguchi Method and they predicted that the Hybrid Composites With 10% Sic by weight shows better tribological properties. By the method of the Taguchi wear test of polymer composites, the wear factor depends on the applied and speed of the rotating disc. Taguchi method proved to be the best method in reducing the number of tests to be conducted. Narayana Murty et al [13] studied the hot working characteristics of SiC and Al_2O_3 particulate reinforced polymer matrix composites.

Park et al [15] investigated the effect of Al_2O_3 in Aluminum by varying the percentage of reinforcement of particles from 5% to 30% and stated that the increase in volume fraction of Al_2O_3 decreases fractures toughness of the developed MMC. The researchers proposed that enhanced properties are due to a decrease in the inter-particle spacing between the nucleated micro-cavities. Park et al [8] investigated the high cycle fatigue behavior of 6061 Al-Mg-Si alloy reinforced Al_2O_3 microspheres with different volume fraction from 5% and 30%. The addition of dry solids such as organic fillers in polymer matrices has enhanced the sustainability of raw materials and make them reclaimable. Madhusudhan T, et al [13] suggested that there is a scope for developing Mechanical Characterization of Jute and Rubber Particles Reinforced Epoxy Polymer Composites.

FILLERS USED IN THE RESEARCH WORK

Molybdenum disulfide is an inorganic compound consisting of molybdenum and sulfur and represented by chemical formula **MoS_2**. It falls under transition metal dichalcogenide. It generally exists as principal oremolybdenite, which is silvery black solid [14]. MoS_2 is not affected by dilute acids and is non-reactive material. The appearance of molybdenum disulfide is similar to graphite, which is widely used as a solid lubricant due to the low coefficient of friction. MoS_2 is preferred lubricating materials it is constituted by the layered structure and has a low coefficient of friction. When a shear stress is applied to the material, it releases energy at the interlayer. Extensive work has been carried out to characterize the coefficient of friction and shear strength of MoS_2 by exposing to different environment [15, 16]. MoS_2 shear strength is proportional to the coefficient of friction which enhances the shear strength due to super lubricity property.

Titanium dioxide (**TiO_2**) is widely used in plastics and related applications, which is a white pigment and is non-opaque to UV radiations. It reduces damages from UV radiations as light scatters by the powder particles having high refractive index [17]. Titanium dioxide is used in ceramic glazers, tattoo pigment and in styptic pencils. Titanium dioxide produced in different particle sizes, which can be dispersible in oil and water.

Aluminumoxide (**Al_2O_3**) is a hard abrasive used in aluminum metal production and is also used as a refractory material due to its high melting point [18]. This is a widely used economical abrasive substituting industrial diamond. Its low heat retention and low specific heat make it widely used in grinding applications such as sandpapers, cutoff tools, and spark plug insulators [19].

TENSILE TESTING

The tensile strength and Young's modulus of developed poly vinyl ester/ glass fiber composites with and without fillers were determined according to ASTM D 638 M-93. The dog-bone-shaped test specimens were cut out from the developed composite laminate. The specimens were tested at a crosshead speed of 2 mm/min and five specimens for each of the composition were tested.

Table 1: Showing the Results of Young's Modulus and Ultimate Tensile Strength of Polyvinyl Ester Composite with Al_2O_3, MoS_2 & TiO_2 Fillers

Sl. No.	Sample Name	Width (mm)	Thickness (mm)	Area (mm^2)	Peak Load (n)	Stress (MPa)	Strain	Young's Modulus (MPa)	Ultimate Tensile Strength (N/mm^2)
1	ve +gf	25.6	4	102.2	4677.9	27.45	0.6109	32.15	45.754
2	ve+gf+al_2o_3 (7.5%)	25.2	3.55	89.425	10352	69.46	0.0581	93.5	85.731
3	ve+gf+al_2o_3 (10%)	25.9	3.73	96.756	9898.9	61.38	0.053	95.4	102.308
4	ve+gf+al_2o_3 (12.5%)	25.2	2.69	67.78	5812	51.43	0.0455	166.94	115.766
5	ve+gf+mos_2 (7.5%)	25	3.14	78.469	10246	78.35	0.072	66.16	130.58
6	ve+gf+mos_2 (10%)	26.5	2.39	63.263	9047.9	85.81	0.085	55.76	143.02
7	ve+gf+mos_2 (12.5%)	26	2.49	64.665	10004	92.82	0.0929	93.58	154.698
8	ve+gf+tio_2 (7.5%)	26.6	3.35	89.077	8463.5	57.01	0.0684	118.67	95.013
9	ve+gf+tio_2 (10%)	25.6	2.76	70.739	8824.3	74.85	0.057	140.09	124.74
10	ve+gf+tio_2 (12.5%)	25.6	2.39	61.136	10471	102.8	0.0649	157.12	171.27

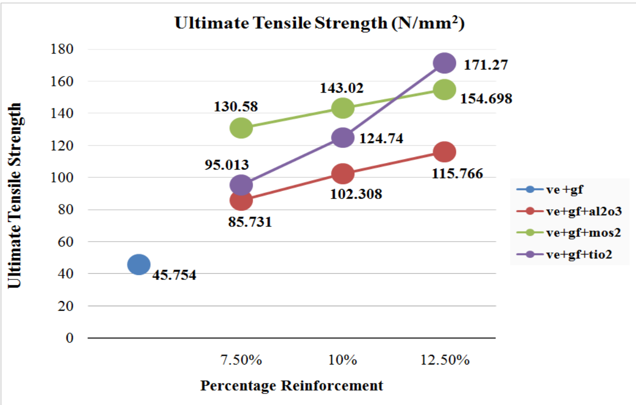

Figure 1: Graph of Ultimate Tensile Strength of Polyvinyl Ester Composite with Al_2O_3, MoS_2 & TiO_2 Fillers

FLEXURAL TESTING

Bending test was carried out for the developed polyvinyl ester/glass fiber composites with and without fillers and ascertained flexural properties as per the ASTM D790M-86. Test Method 1 of ASTM D790M-86 was used in which a three point loading system with center loading on a simply supported beam was employed. Specimens were machined from laminates and tested at crosshead speed of 1 mm/min. The test specimen cut is of 90 mm × 12 mm × 3 mm. Five specimens for each of the compositions were tested on a fully automated Kalpak-100Kuniversal testing machine

connected to a computer which was aided by KALPAK software. A 50-kN load cell was used.

The figure 1 shows at the most improvement in UTS of filled polyvinyl ester composite by 50 to 60 %. The addition of TiO_2 filler has better strength compared to other fillers and maximum UTS was observed for 12.5% Tio_2 and there is a drastic increase in tensile strength to almost 40% compared with 7.5% Tio_2 polyvinyl ester composite. The increase in % of filler has enhanced the ultimate strength monotonously except for 12.5% of Tio_2 filler in the polyvinyl ester/GF composites. This may be due to improvements in the interfacial bonding of fiber with the polyvinyl ester matrix, which may be due to improvement in the rough fiber surface due to filler embodiment on the surface of the fiber.

The figure 2 shows improvement in Young' s modulus of filled polyvinyl ester composite by 50 to 60 %. This may be due to improvements in the interfacial bonding of fiber with the polyvinyl ester matrix, which may be due to improvement in the rough fiber surface due to filler embodiment on the surface of the fiber. The addition of Al_2O_3 filler has better Young' s modulus compared to other fillers and maximum Young' s modulus was observed for 12.5% Al_2O_3 and there is a drastic increase in Young' s modulus to almost 40% compared with 7.5% Al_2O_3 polyvinyl ester composites. The increase in % of filler has enhanced the ultimate strength monotonously except for 10% of MoS_2 filler in the polyvinyl ester/GF composites.

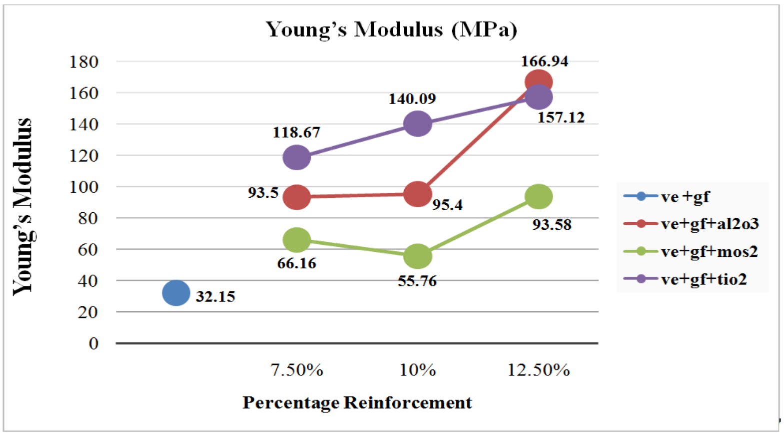

Figure 2: Graph of Young's Modulus of Polyvinyl Ester Composite with Al_2O_3, MoS_2 & TiO_2 Fillers

Table 2: Showing the Results of Flexural Modulus and Ultimate Flexural Strength of Polyvinyl Ester Composite with Al_2O_3, MoS_2& TiO_2 Fillers

Sl. No.	Sample Name	Width (mm)	Thickness (mm)	Peak Load (n)	Flexural Stress (MPa) $3pl/2bt^2$	Flexural Modulus (MPa) $L^3m/4bt^3$	Stress (MPa)	Strain (MPa)
1	ve +gf	14.36	4.02	178.95	69.401	4059.48	1.86	0.02958
2	ve+gf+al_2o_3 (7.5%)	13.19	3.16	166.72	113.92	3990.67	2.4	0.053
3	ve+gf+al_2o_3 (10%)	12.73	3.07	222.23	166.702	7212.84	3.412	0.05867
4	ve+gf+al_2o_3 (12.5%)	3.21	3.21	246.52	167.3	12462	3.58	0.05422
5	ve+gf+mos_2 (7.5%)	13.67	2.54	110.28	112.539	2639.36	1.9	0.0511
6	ve+gf+mos_2 (10%)	13.83	2.35	122.12	143.904	6383.09	2.254	0.05373
7	ve+gf+mos_2 (12.5%)	13.65	2.32	128.8	157.779	2397.92	2.069	0.04657
8	ve+gf+tio_2 (7.5%)	12.95	4	108.66	118.74	9809.16	3.167	0.049
9	ve+gf+tio_2 (10%)	12.7	3.44	249.47	149.39	5594.21	3.42	0.05135
10	ve+gf+tio_2 (12.5%)	13.03	2.16	273.38	160.86	3427.17	2.316	0.058

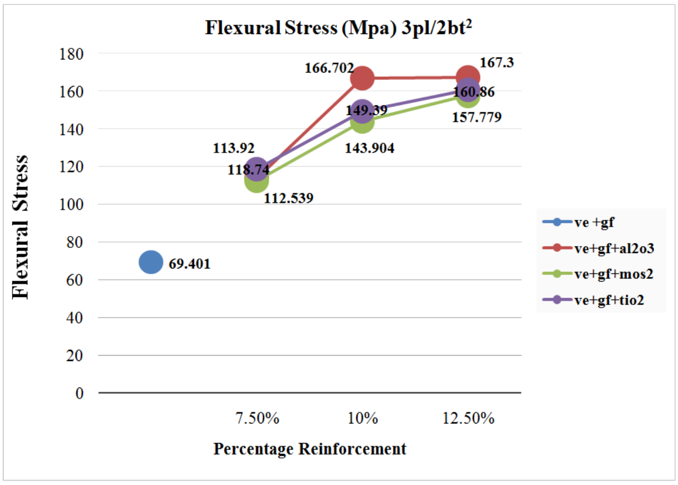

Figure 3: Graph of Flexural Strength of Polyvinyl Ester Composite with Al_2O_3, MoS_2& TiO_2 Fillers

Figure 3 shows the flexural strength and modulus of 7.5 wt.% Al_2O_3 filled improved by 48% compared to that of unfilled poly-vinyl ester composite. However, the effect of MoS_2 filler loading is more significant and the enhancement in flexural strength and modulus by 60% for 7.5 wt.% loading in polyvinyl ester composite. Variation of flexural strength for different filler type and loading (see online version for colors), the effect of filler loading on the variation of flexural strength behavior is shown in Figure 3. All the fillers showed improvement in the flexural strength of the polyvinyl ester composite. These particles change the fiber surface by increasing surface roughness and provide strong bonding between the fiber/matrix interface causing changes in thermal residual stresses at the fiber surface. Flexural strength of particulate filled polyvinyl ester composite composites improved due to strong interfacial adhesion and the surface roughness act as mechanical interlocking.

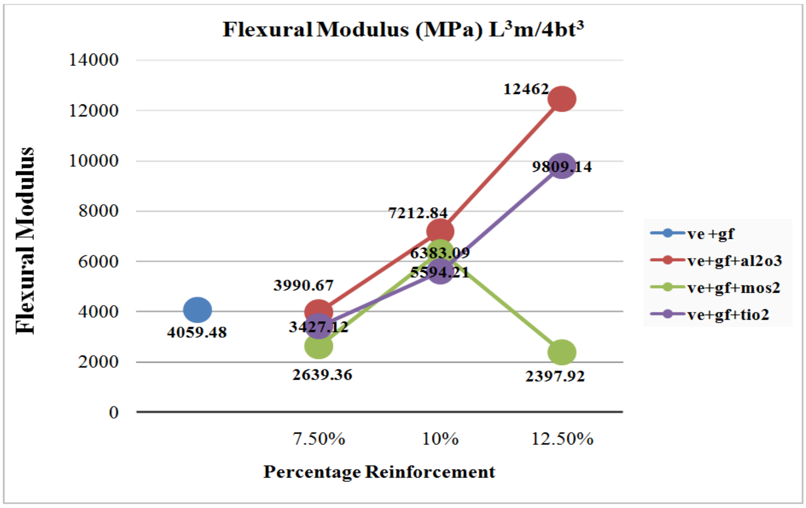

Figure 4: Graph of Flexural Modulus of Polyvinyl Ester Composite with Al_2O_3, MoS_2& TiO_2 Fillers

The influence of filler loading on the flexural modulus is presented in Figure4. From figure 4 it is observed that flexural modulus was improved by incorporating Al_2O_3 and MoS_2 fillers in polyvinyl ester/GF composite. It is also evident that the addition of MoS_2 filler is more beneficial than that of Al_2O_3 filler in improving the flexural strength and modulus

of polyvinylester/GF composite. This result can be interpreted as follows. The flexural modulus is a material constant, which depend on the slope of the first linear portion of the load-deflection curve. The constituents of polyvinyl/GF composite have the same elastic deformation in the load-deflection curve. High stiffness and high strength of Al_2O_3 and MoS_2 particles under low loads cannot synchronize with the deformation of fiber and matrix and promotes micro-cracks in the matrix above or below the particles and which propagated parallel to the fiber direction. Therefore, the modulus of unfilled is less than that of particulate filled polyvinyl ester/GF composites. The improvement in the flexural strength and modulus of particulate filled poly vinyl ester/GF composites were due to the enhancement in the interfacial bond strength between the micron-phased matrix and the glass fiber. The reduction in modulus and flexural strength of MOS_2 composites as filler percentage increasing from 10% to 12.5% is due to, molybdenum disulfide with a low coefficient of friction which is similar to graphite used widely as the solid lubricant. MoS_2 is preferred lubricating material (see below) as it is constituted by the layered structure and has a low coefficient of friction.

Fractography of Tensile Test Filled Polyvinyl Ester/GF Hybrid Composites

Tensile fractured surfaces were subjected to Scanning Electron Microscopy (SEM). Fractured surfaces of unfilled polyvinyl ester/ GF, Al_2O_3 filled and MoS_2filled polyvinyl ester/GF hybrid composites are shown in Figures 5(a), 5(b) and 5(c) respectively. For unfilled polyvinyl ester/GF had extensive matrix damage observed during tensile testing. Few rivers marks are evident from the micrograph [Figure 5(a)], which are steps between cleavage lines and are expected to propagate in the multi-direction from the point of crack initiation [Figure 5(a)].

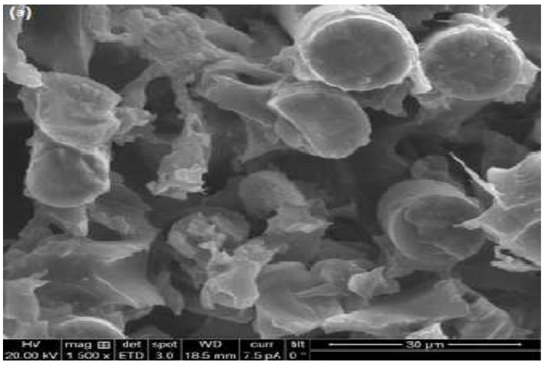

Figure 5(a): Showing Scanning Electron Micrograph of Polyvinyl Ester/GF Composite

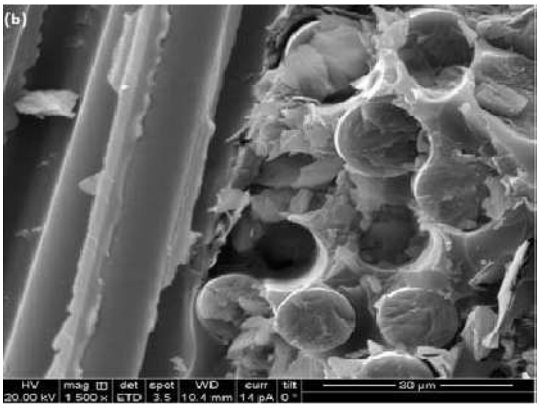

Figure 5(b): Showing Scanning Electron Micrograph of Polyvinyl Ester/GF with Al_2O_3 Filler Composite

Al_2O_3 filled polyvinyl ester/GF composite, it is observed that there are few fiber pull-out and found better adhesion of Al_2O_3 and polyvinyl ester matrix which is evident from the SEM shown in Figure 5(b). Under the tensile loading conditions, most particles do not fracture which is evident Figure 5(b), which is obtained on the tensile fracture surface of Al_2O_3 filled poly-vinyl ester/GF specimen shown in Figure 5(b), the researchers observed matrix cracks, particles pull-out, debonding of filler-matrix and a significant step appearance of fiber pulled-out regions.

Figure 5(c) shows the tensile fractured surface of MoS_2 filled poly vinyl ester/GF composite. From the figure, it is observed that the polyvinyl ester matrix failure starts at the weak interface and due to the good bonding of fiber and filler with the matrix, a fracture in different planes are formed behind fibers. The presence of fibers contributes to the formation of additional step structures from which new surfaces were formed on flexural strength and a flexural modulus of polyvinyl ester/GF composites.

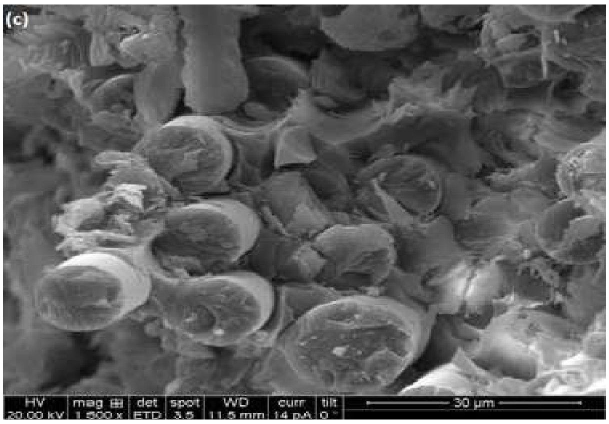

Figure 5(c): Showing Scanning Electron Micrograph of Polyvinyl Ester/GF with MoS_2 Filler Composite

CONCLUSIONS

- The hybrid composite of polyvinyl ester/Glass Fiber with Alumina (Al_2O_3), Molybdenum disulfide (MoS_2) and Titanium oxide (TiO_2) as fillers were successfully fabricated.
- The hybrid composites developed were subjected to tensile and flexural tests and results are tabulated along with the graphs
- The increase in % of filler has enhanced the ultimate strength monotonously except for 12.5% of Tio_2 filler in the polyvinyl ester/GF composites. This may be due to improvements in the interfacial bonding of fiber with the polyvinyl ester matrix.
- The addition of TiO_2 filler has better strength compared to other fillers and maximum UTS was observed for 12.5% Tio_2 and there is a drastic increase in tensile strength to almost 40% compared with 7.5% Tio_2 polyvinyl ester composite.
- The addition of Al_2O_3 filler has better Young’s modulus compared to other fillers and maximum Young’s modulus was observed for 12.5% Al_2O_3 and there is a drastic increase in Young’s modulus to almost 40% compared with 7.5% Al_2O_3 polyvinyl ester composite.

- All the fillers showed improvement in the flexural strength of the polyvinyl ester composite composites.
- The reduction in modulus and flexural strength of MOS_2 composites as filler percentage increasing from 10% to 12.5% is due to, molybdenum disulfide with a low coefficient of friction.
- River marks are evident from the micrograph which are steps between cleavage lines and are expected to propagate in the multi-direction from the point of crack initiation.

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